

ANALYST:		VPDES NO	
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Parameter: Total Kjeldahl Nitrogen  
Method: Digestion followed by Distillation  
04/01

METHOD OF ANALYSIS:

	18th Edition of Standard Methods 4500N <sub>org</sub> B/C
	Methods for Chemical Analysis of Water and Wastes 351.3
	ASTM D3590-89(A)

	Y	N
1) Is digestion unit capable of reaching 365° to 370°C? [SM-2.a]		
2) Are clean glass beads or boiling chips added to the digestion flask? [SM-B.4.b, C.4.c]		
3) Are digestion fumes removed by a fume hood or evacuation equipment? [SM-4.c]		
4) Is ammonia free water used in all aspects of the procedure? [SM-3; 351.3-7.1]		
5) Are all reagents prepared in accordance with the selected approved method? [SM-3; 351.3-7]		
6) <b>Macro:</b> Is 50 mL of sulfuric acid-potassium sulfate solution and 10 mL of copper sulfate added to 500 mL of sample (or 500 mL of diluted sample)? <b>Micro:</b> Is 10 mL of digestion reagent plus 2 mL of copper sulfate added to 50 mL of sample (or 50 mL of diluted sample)? [SM-A.4; EPA approved for 351.3]		
7) Is a blank carried through each digestion series? [SM-B.4.f, C.4.e; 351.3-8.4]		
8) Are samples digested for an additional thirty minutes after copious white fumes are generated? [SM-4.c; 351.3-8.2.1 or 8.3.1]		
9) Are samples prevented from being heated to dryness? [Permit]		
10) Is the digestate allowed to cool to room temperature before proceeding? [SM-4.c; 351.3-8.2.1 or 8.3.1]		
11) Is digestate diluted to approximately 300 mL with ammonia free water (30 mL for micro-digestions)? [SM-4.c; 351.3-8.2.1 or 8.3.1]		
12) Is 50 mL for SM, 100 mL for 351.3 (10 mL for micro.) of hydroxide-thiosulfate reagent carefully added to the diluted digestate to form an alkaline layer at the bottom of the? [SM-4.c; 351.3-8.2.2 or 8.3.2]		
13) Is ammonia distillation apparatus constructed of borosilicate glass with ground glass or TFE joints/connections (rubber or plastic tubing is to be avoided)? [SM-2.b; 351.3-6.2]		
14) Are the flasks contents mixed thoroughly after connection to the distillation apparatus? [SM-4.c; 351.3-8.2.2 or 8.3.2]		
15) Is 200 mL for SM, 300 mL for 351.3 (30 to 40 mL for micro.) of distillate collected? [SM-4.d; 351.3-8.2 or 8.3.1]		
16) Is the distillation apparatus steamed out with a solution of borate buffer and reagent water adjusted to a pH 9.5 SU (1:1 mixture of hydroxide/thiosulfate solution for EPA 351.3) immediately before each use? [SM-4500-NH3 B.4.a; 351.3-8.1]		
17) Is the distillation apparatus kept assembled until samples are distilled? [SM-4500-NH3 B.4.c]		
18) Is the boric acid used in the collection flask? [SM-4.d; 351.3-8.2.4 or 8.3.3]		
19) Is the distillation carried out at a rate of 6 to 10 mL/min? [351.3-8.3.4]		
20) Does the delivery tube of the distillation apparatus extend below the surface of the receiving solution? [SM-4.d; 351.3-8.2.3 or 8.3.3]		
21) Is an approved ammonia nitrogen procedure used for TKN determination? [SM-4.e; 351.3-8.2.5 or 8.3.5]		

PROBLEMS:

TKN dig-dist

